

(12) **UK Patent Application** (19) **GB** (11) **2 248 853 A** (13)
(43) Date of A publication 22.04.1992

(21) Application No **9120561.7**

(22) Date of filing **27.09.1991**

(30) Priority data

(31) **9021042**
9021976

(32) **27.09.1990**
09.10.1990

(33) **GB**

(71) Applicants

Anthony Copeland Jones

**Rose Tree Cottage, Georgian Close, Eccleston Park,
Prescot, Merseyside, L35 7JW, United Kingdom**

Graham Williams

**Tinkersdale, Tower Road North, Heswall, Wirral,
United Kingdom**

Albert Barry Leese

**14 Lakeside, Lodge Road, Alsager, Cheshire,
United Kingdom**

(72) Inventors

Anthony Copeland Jones

Graham Williams

Albert Barry Leese

(51) INT CL⁵
C23C 18/10

(52) UK CL (Edition K)

C7F FHD FR841 F782 F801
U1S S1415 S1917 S3037

(56) Documents cited

GB 1178954 A SU 0971913 A US 4144360 A

(58) Field of search

UK CL (Edition K) C7F FHB FHD FHZ
INT CL⁵ C23C
Online databases: WPI, CLAIMS

(74) Agent and/or Address for Service

Roystons

**Tower Building, Water Street, Liverpool, L3 1BA,
United Kingdom**

(54) **Coating of substrates with aluminium using an alane adduct**

(57) A method of coating a substrate such as glass with aluminium to form a mirror comprises the steps of forming a solution of an alane amine adduct and depositing same on a heated substrate, whereby the adduct decomposes to leave a coating of elemental aluminium. The alane adduct preferably has the formula $AlH_3(NR'R''R''')$, where n is 1 or 2 and R', R'', R''' are selected from alkyl, aryl, alkaryl, or aralkyl radical, eg trimethylamine alane, bis-(trimethylamine) alane, and dimethylethylamine alane. Amines, ethers and aryl compounds may be used as the solvent to dissolve the aluminium compound. In the case of a liquid alane adduct, such as dimethylethylamine alane, it may not be necessary to form a solution thereof prior to coating. After coating a sealer layer may be applied to inhibit oxide tunnelling.

GB 2 248 853 A

Title: Coating of substrates

DESCRIPTION

This invention concerns formation of a coating of aluminium on a substrate and, in particular, concerns a
5 method of producing mirrors.

Mirrors are mostly produced by forming a silver coating on one surface of a sheet of glass, the silver coating acting as a reflector. The production of such mirrors is relatively expensive due to the use of
10 silver.

One object of this invention is to provide an alternative to silver for mirror production.

Another object of this invention is to provide a method of coating aluminium onto a substrate, such as
15 glass.

According to one aspect of the invention there is provided a method of coating a substrate with a layer of aluminium comprising the steps of forming a solution of an alane adduct and depositing the solution onto a
20 heated substrate.

According to another aspect of the invention there is provided a substrate coated with a layer of aluminium by the method of depositing on the heated substrate a solution of an alane adduct.

A preferred use of the invention is in the production of mirrors where the substrate is glass and the aluminium coating forms a reflective layer. It is envisaged that the invention may be used in conjunction
 5 with float glass production, wherein aluminium deposition is carried out on hot glass, typically at 180°C, emerging from the float glass process. It is, in fact, preferred that any substrate used in the invention be heated to at least 100°C so as to drive off moisture
 10 from the substrate and/or atmosphere that could otherwise cause oxidation of the aluminium.

The deposition of aluminium on a substrate is preferably carried out in an inert atmosphere, such as of nitrogen or argon. Although the aluminium deposition
 15 may be carried out in an atmosphere containing oxygen, that is less desirable due to the risks of oxidation and fire particularly with the presence of solvent and the use of high temperatures.

20 After coating a substrate with aluminium, a sealer layer may be applied to the aluminium coating for protection and to inhibit oxide tunnelling.

The aluminium compound used in the invention is preferably an alane adduct, especially with a triamine.

~~The aluminium adduct preferably has the formula AlH_3~~
 25 $(NR_3)_n (NR'R''R''')_n$ where n is 1 or 2 and R', R'' and R''' are selected from alkyl, aryl, alkaryl or aralkyl

radical. Particularly suitable alane adducts for use in the present invention are believed to be trimethylamine alane, bis-(trimethylamine) alane and dimethylethylamine alane.

5 The solvent used to dissolve the aluminium compound is preferably a donor solvent. Examples of suitable donor solvents include amines, ethers and aryl compounds. Of amines, tertiary amines are preferred and especially those of the formula $N(C_n H_{2n+1})_3$, where n is
10 from 1 to 12, such as tri-n-butylamine and triethylamine. Triaryl amines, trialkaryl amines and triaralkyl amines may also be used as solvents for the aluminium compound. In the case of a liquid alane adduct, such as dimethylethylamine alane, it may not be
15 necessary to form a solution thereof prior to coating.

Of ethers, dialkyl ethers are preferred and especially those of the formula $(C_n H_{2n+1})_2O$, where n is from 1 to 12. Diaryl ethers, dialkaryl ethers, diaralkyl ethers and cyclic ethers as well as mixed
20 ethers may also be used as solvents for the aluminium compound. Examples of suitable ethers include diethyl ether, diphenyl ether and tetrahydrofuran.

Of aryl, compounds benzene and toluene may be suitable solvents for the aluminium compound.

25 The amount of aluminium compound used in the invention will be preferably sufficient to provide a

solution having upto 20%, preferably 5 to 10%, by weight aluminium content.

The solution of aluminium compound may be deposited on the substrate in any suitable way. Preferably the aluminium compound solution will be dropped onto the substrate, the substrate and deposition head(s) moving relative to each other to form a relatively thin even coating on the substrate. Under the influence of heat the aluminium compound decomposes to leave a coating of elemental aluminium on the substrate. That coating when the substrate is glass can provide a reflective surface that is of reasonable mirror quality but at a lower cost than a silver coating.

This invention will now be further described by means of the following Example.

Example

10g of trimethylamine alane were dissolved in 29g of trisbutylamine. The resultant solution was applied to a glass substrate heated to 180°C in a nitrogen atmosphere. The glass thus received a reflective coating of aluminium to form a mirror. A coat of sealer was applied to the aluminium coating to inhibit oxide tunnelling.

CLAIMS

1. A method of coating a substrate with a layer of aluminium comprising the steps of forming a solution of an alane adduct and depositing the solution onto a heated substrate.

2. A method as claimed in claim 1, wherein the substrate is glass.

3. A method as claimed in claim 1 or 2, wherein the substrate is heated to at least 100°C.

4. A method as claimed in claim 1, 2 or 3, wherein deposition is carried out in a substantially inert atmosphere.

5. A method as claimed in any one of claims 1 to 4, further comprising the step of applying a sealer layer to the aluminium coating.

6. A method as claimed in any one of claims 1 to 5, wherein the alane adduct is an alane adduct with a triamine.

7. A method as claimed in claim 6, wherein the alane adduct has the formula $\text{AlH}_3 \cdot (\text{N R}' \text{ R}'' \text{ R}''')_n$, wherein n is 1 or 2 and R' , R'' and R''' are selected from alkyl, aryl, alkaryl and aralkyl radicals.

8. A method as claimed in claim 7, wherein the alane adduct is selected from trimethylamine alane, bis-(trimethylamine) alane and dimethylethylamine alane.

9. A method as claimed in any one of claims 1 to 8, wherein the solvent is a donor solvent.

10. A method as claimed in claim 9, wherein the solvent is selected from amines, ethers and aryl
5 compounds.

11. A method as claimed in claim 10, wherein the solvent is a tertiary amine.

12. A method as claimed in claim 11, wherein the tertiary amine is of the formula $N(C_n H_{2n+1})_3$, where n is
10 from 1 to 12.

13. A method as claimed in claim 12, wherein the tertiary amine is tri-n-butylamine or triethylamine.

14. A method as claimed in claim 10, wherein the solvent is a dialkyl ether.

15 15. A method as claimed in claim 14, wherein the ether has the formula $(C_n H_{2n+1})_2O$, where n is from 1 to 12.

16. A method as claimed in claim 10, wherein the solvent is selected from diethyl ether, diphenyl ether
20 and tetrahydrofuran.

17. A method as claimed in claim 10, wherein the solvent is selected from benzene and toluene.

18. A method as claimed in any one of claims 1 to 17, wherein the amount of aluminium compound used is
25 sufficient to provide a solution having upto 20% by weight aluminium content.

19. A method as claimed in claim 18, wherein the amount of aluminium compound used is sufficient to provide a solution having from 5 to 10% by weight aluminium content.

5 20. A method as claimed in any one of claims 1 to 19, wherein the aluminium compound solution is dropped onto a moving substrate.

21. A method of coating a substrate substantially as hereinbefore described with reference to the foregoing
10 Example.

22. A substrate coated by a method as claimed in any one of claim 1 to 21.

Patents Act 1977
 Examiner's report to the Comptroller under
 Section 17 (The Search Report)

- 8 -

Application number

9120561.7

Relevant Technical fields

(i) UK Cl (Edition K) C7F (FHB, FHD, FHZ)

(ii) Int Cl (Edition 5) C23C

Search Examiner

P G BEDDOE

Databases (see over)

(i) UK Patent Office

(ii) ONLINE DATABASES: WPI, CLAIMS

Date of Search

26 NOVEMBER 1991

Documents considered relevant following a search in respect of claims

1-22

Category (see over)	Identity of document and relevant passages	Relevant to claim(s)
X	GB 1,178,954 A (CONTINENTAL) see especially Table at page 4	1,3
X	US 4,144,360 A (SIEMENS) see especially Claim 2 and Example 13	1,2,6,7, 8
X	SU 971913 A (TAMBOV) see English language abstract (WPI Acc. No: 83-75771/36)	1,3,4,6, 7,8

Category	Identity of document and relevant passages	Relevant to claim(s)

Categories of documents

X: Document indicating lack of novelty or of inventive step.

Y: Document indicating lack of inventive step if combined with one or more other documents of the same category.

A: Document indicating technological background and/or state of the art.

P: Document published on or after the declared priority date but before the filing date of the present application.

E: Patent document published on or after, but with priority date earlier than, the filing date of the present application.

&: Member of the same patent family, corresponding document.

Databases: The UK Patent Office database comprises classified collections of GB, EP, WO and US patent specifications as outlined periodically in the Official Journal (Patents). The on-line databases considered for search are also listed periodically in the Official Journal (Patents).